Pyriproxyfen/129032/Valent USA Corporation
OPPTS 860.1500
Crop Field Trial - Cantaloupe

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Registration Action Branch 1
(RAB1)
Health Effects Division
(HED) (7509C)

STUDY REPORT:

MRID No. 455560-04 C.A. Green (2001) Magnitude of the Residues of Pyriproxyfen on Cantaloupe. Laboratory Project Identification Number: 20204. Unpublished study prepared by Valent USA Corporation. 186 pages.

EXECUTIVE SUMMARY:

Supervised crop field trials were conducted in Arizona (2 trials), California (2 trials), North Carolina, and Texas in/on cantaloupe treated two times at an application rate of 0.066 lb ai/A (seasonal rate of 0.132 lb a.i./A) with a pre-harvest interval (PHI) of 7 days and a retreatment interval (RTI) of 14 days. The results from these trials show that pyriproxyfen residues ranged from <0.01-0.04 ppm in/on treated cantaloupe samples. Pyriproxyfen residues demonstrated a slight decline with increasing PHI over a range of 3-14 days. Doubling the application rate resulted in an increase in the pyriproxyfen residue level. However, the PHI and application rate trends were based on the results of just one trial each. Evidence was also provided showing that pyriproxyfen residues predominate on the surface of cantaloupe. The limit of quantitation (LOQ) was established at 0.01 ppm.

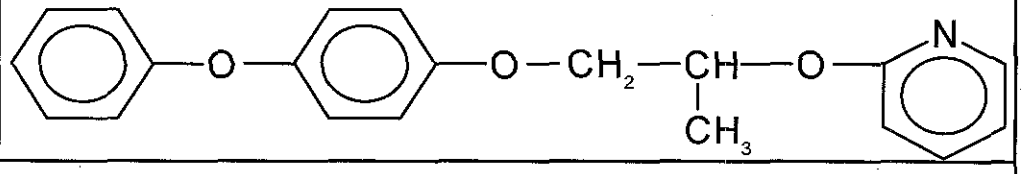
COMPLIANCE:

Signed and dated Good Laboratory Practices (GLP), Quality Assurance and Data Confidentiality statements were provided. Several minor GLP deviations were noted, although they did not impact the validity of the study.

A. BACKGROUND INFORMATION

Pyriproxyfen is an analogue of an insect juvenile hormone and interferes with the hormonal control of insect growth and development, thereby inhibiting egg hatch, larval embryogenesis, metamorphosis, and adult emergence. There are currently three end-use products of pyriproxyfen with food/feed uses that are registered to Valent: two emulsifiable concentrates (EC) and a wettable powder (WP). These formulations are registered for use on bushberry, citrus fruits, cotton, fruiting vegetables, guava, lychee, pome fruits, stone fruits, and tree nuts, and are marketed under the trade names KNACK® Insect Growth Regulator [0.86 lb/gal EC; EPA Reg. No. 59639-95], ESTEEM® Insect Growth Regulator [2.9 lb/gal EC; EPA Reg. No. 59639-104], and ESTEEM® 35 WP Insect Growth Regulator [35% WP; EPA Reg. No. 59639-115].

TABLE A.1. Test Compound Nomenclature

Compound	Chemical Structure
$C_{20}H_{19}NO_3$	
Common name	Pyriproxyfen
IUPAC name	4-phenoxyphenyl (RS)-2-(2-pyridyloxy)propyl ether
CAS name	2-[1-methyl-2-(4-phenoxyphenoxy)ethoxy]pyridine
CAS #	95737-68-1
End-use product/EP	Knack IGR, Esteem IGR

B. EXPERIMENTAL DESIGN

B.1. Study Site Information

No extreme weather events were noted at any of the field trial sites involved in the present study. Where necessary, irrigation was provided.

TABLE B.1.1. Study Use Pattern.

Location (City, State/Year)	EP[1]	Application						Tank Mix Adjuvants
		Timing	Rate, lb a.i./A	RTI (days)	Treat. No.	Method[2]	Total Rate, lb a.i./A	
Willacy County, TX/1999	Knack IGR (0.86 EC)	1) 21 (±1) days before harvest 2) 7 (±1) days before harvest	1) 0.0677 2) 0.0681	14	2	tractor-mounted boom sprayers	0.136	None
Madera County, CA/1999	Knack IGR (0.86 EC)	1) 21 (±1) days before harvest 2) 7 (±1) days before harvest	1) 0.0688 2) 0.0677	14	2	tractor-mounted boom sprayers	0.136	None
Pinal County, AZ/1999	Knack IGR (0.86 EC)	1) 21 (±1) days before harvest 2) 7 (±1) days before harvest	1) 0.0659 2) 0.0661	14	2	tractor-mounted boom sprayers	0.132	None
Martin County, NC/2000	Knack IGR (0.86 EC)	1) 21 (±1) days before harvest 2) 7 (±1) days before harvest	1) 0.0666 2) 0.0675	14	2	tractor-mounted boom sprayers	0.134	None
Pepin County, WI/2000	Knack IGR (0.86 EC)	1) 21 (±1) days before harvest 2) 7 (±1) days before harvest	1) 0.0668 2) 0.0677	14	2	tractor-mounted boom sprayers	0.134	None
Madera County, CA/2000	Knack IGR (0.86 EC)	1) 21 (±1) days before harvest 2) 7 (±1) days before harvest	1) 0.0670 2) 0.0666	14	2	tractor-mounted boom sprayers	0.134	None

Pinal County, AZ/2000	Knack IGR (0.86 EC)	1) 21 (±1) days before harvest 2) 7 (±1) days before harvest	1) 0.0679 2) 0.0657	14	2	tractor-mounted boom sprayers	0.134 (1x)	None
			1) 0.142 2) 0.128				0.270 (2x)	

[1] EP = End-use Product
[2] Trials utilized 8.4-10.8 gallons diluted spray per acre.

TABLE B.1.3. Trial Numbers and Geographical Locations

Crop		US Growing Regions													Total trials
		1	2	3	4	5	6	7	8	9	10	11	12	13	
Cantaloupe	Submitted		1			1	1				4				7
	Requested		1			1	1				3				6

B.2. Analytical Methodology

The samples were analyzed using the reference method: "Determination of Pyriproxyfen Residues in Apples, Pears, and Citrus Fruits", Valent method RM-33P-1-3a. In brief, residues of pyriproxyfen were extracted from samples with acetone, partitioned with dichloromethane/water, and cleaned using silica gel column chromatography. Quantitation for pyriproxyfen was by gas chromatography with a nitrogen-phosphorus detector (GC/NPD). The limit of quantitation (LOD) was established at 0.01 ppm; the limit of detection (LOD) was not estimated.

C. RESULTS AND DISCUSSION

The number of trials and the geographic representation are both adequate for cantaloupe as a representative commodity of crop group 9, where six field trials are requested (OPPTS 860.1500, Table 2). Detectable levels of pyriproxyfen residues were found in most of the treated cantaloupe samples, although these levels were all ≤ 0.04 ppm. The residue decline data submitted indicates that pyriproxyfen residues show a slight decrease when the PHI is extended from 3 to 14 days. Doubling the application rate appeared to result in an increase in the pyriproxyfen residue level. However, the PHI and application rate trends were based on the results of just one trial each.

Concurrent recovery results indicate that the data collection method is adequate for detecting pyriproxyfen residues in/on cantaloupe samples (see Table C.1). Satisfactory recovery values were obtained from cantaloupe samples spiked at 0.02 and 0.10 ppm. All untreated control samples were free of pyriproxyfen residues and interferences, except for one untreated control sample where 0.003 ppm pyriproxyfen was detected.

The longest harvest-to-analysis interval of any field treated sample was 13 days (Table

C.2). Thus, a complete storage stability study was not conducted. However, the results of a limited storage stability study demonstrated that pyriproxyfen residues are stable in cantaloupe when stored frozen for 12 days.

As shown in Tables C.3 and C.4, pyriproxyfen residue levels in cantaloupe samples ranged from <0.01-0.04 ppm, when the PHI and RTI were 7 and 14 days, respectively. Table C.4 also shows the effect of PHI on pyriproxyfen residue levels in/on cantaloupe samples when the PHI is incremented over a range of 3-14 days. The residue levels show a slight decline over time.

Studies of pyriproxyfen residues in cantaloupe rinds versus centers in fruits from the 2000 trial in CA demonstrated that the pyriproxyfen residues were on the surface of the fruit.

TABLE C.1. Summary of Concurrent Recoveries of Pyriproxyfen from Cantaloupe.

Matrix	Analyte	Spike level (mg/kg)	Sample size (n)	Recoveries (%)	Mean ± std dev
Cantaloupe	Pyriproxyfen	0.020	9	73-103	93 ± 9
		0.10	10	81-102	93 ± 6

TABLE C.2. Summary of Storage Conditions

Matrix (RAC or Extract)	Storage Temp. (°C)	Actual Storage Duration (days)	Limit of Demonstrated Storage Stability (days)
Cantaloupe	<0	13*	12

* One untreated cantaloupe control sample was extracted 20 days after harvest.

TABLE C.3. Residue Data from Crop Field Trials with Pyriproxyfen.

Location (City, State/Year)	Region	Crop/ Variety	Application Rate, lbs ai/A	Total Rate, lbs ai/A	RTI (days)	PHI (days)	Residues (ppm)
Willacy County, TX/1999	6	Cantaloupe/ Tam Uvalde	0.0677 0.0681	0.136	14	7	0.04, 0.03
Madera County, CA/1999	10	Cantaloupe/ Hales Jumbo	0.0688 0.0677	0.136	14	7	0.02, 0.03
Pinal County, AZ/1999	10	Cantaloupe/ Hy Mark	0.0659 0.0661	0.132	14	3	0.02, 0.02
						7	0.01, 0.02
						10	0.01, <0.01

						14	<0.01, <0.01
Martin County, NC/2000	2	Cantaloupe/ Ambrosia Hybrid 30510	0.0666 0.0675	0.134	14	7	0.01, 0.02
Pepin County, WI/2000	5	Cantaloupe/ Primo Hybrid	0.0668 0.0677	0.134	14	7	0.01, 0.01
Madera County, CA/2000	10	Cantaloupe/ Top Mark	0.0670 0.0666	0.134	14	7	0.02, 0.02
Pinal County, AZ/2000	10	Cantaloupe/ Gold Rush	0.0679 0.0657	0.134	14	7	<0.01, <0.01
			0.142 0.128	0.270 (2x)			0.02, 0.02

TABLE C.4. Summary of Residue Data from Crop Field Trials with Pyriproxyfen.

Commodity	Total Applic. Rate, lb a.i./A	PHI (days)	Analyte	Residue Levels (ppm)				
				Min.	Max.	HAFT*	Mean	Std. Dev.
Cantaloupe	0.132	7	Pyriproxyfen	<0.01	0.04	0.035	0.019	0.009

* HAFT = Highest Average Field Trial.

D. CONCLUSION

The crop field trials for cantaloupe (a muskmelon) are classified as acceptable and satisfy the guideline requirements for crop field trials (Residue Chemistry Guidelines OPPTS 860.1500) as they apply to "Vegetable, cucurbit, group 9."

E. STUDY DEFICIENCIES/CLARIFICATIONS

None.